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OBITUARY.

We greatly regret to record the death, on July 23, of Sir William Ramsay, K.C.B., F.R.S., an honorary member of the Society. An obituary notice will appear in a forthcoming issue of the Journal.

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EXCRETION AND SECRETION OF SALVARSAN AND NEO-SALVARSAN.

By JOHN WEBSTER, F.I.C.

(*Read at the Meeting, May* 3, 1916.)

Salvarsan ("606") (or Kharsivan, as the English preparation is termed) and Neo-Salvarsan (or Neo-Kharsivan) are at the present time probably the two most effectual remedies for the treatment of syphilis. With regard to the evolution of these valuable drugs, it may be stated that the goal which the experimenters had in view was a drug that would be non-toxic to the patient, though powerfully toxic as to the parasite (*Spirochæta pallida*)—that is, parasitotropic, but non-organotropic.

After much experimentation it was found that compounds of arsenic with the benzene nucleus gave good results.

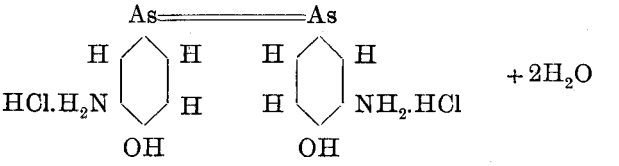
Atoxyl (sodium paramino-phenyl arsenic acid) $NH_2.C_6H_4.As\begin{matrix}OH\\ =O\\ ONa\end{matrix}$ was such a compound. This drug gave apparently most promising results, as its action on the parasite was strong, and it had a low organotropic action. But it was eventually found that atoxyl had not (at any rate in all cases) so low an organotropic action as had been hoped. It had occasionally a marked action on the optic nerve, causing in some cases total blindness.

With regard to atoxyl, it is important to note that it was originally supposed to have the following formula: $C_6H_5.NHAs\begin{matrix}OH\\ =O\\ ONa\end{matrix}$, that is to say, the amino group was directly bound to the arsenic. It was after it had been proved that the amino group

was not directly bound to the arsenic that various substitution products, such as salvarsan, were evolved.

Salvarsan ("606") was found by Ehrlich and his assistant Bertheim to be the most satisfactory compound at the time. This compound (diamino-dihydroxy-arseno-benzene-dihydrochloride), obtained from atoxyl, was proved to have very high parasitotropic action, but very low organotropic action, and to have no such effect on the optic nerve as was the case with atoxyl.

The formula of salvarsan was originally stated to be $C_{12}H_{12}N_2O_2As_2.2HCl$, requiring 34 per cent. of arsenic. Gaebel, however (*Arch. Pharm.*, 1911, **249**, 241-247; *Apoth. Zeit.*, 1911, 215), has shown that the correct formula of salvarsan is $C_{12}H_{12}N_2O_2As_2.2HCl + 2H_2O$, requiring 31·6 per cent. of arsenic. Graphically it may be expressed thus:

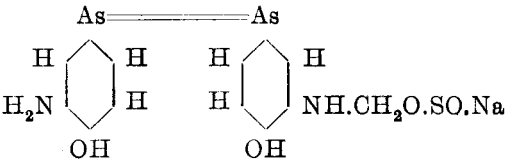


Chemical Tests.—Salvarsan readily gives the Reinsch test. The statement that salvarsan responds to the Marsh test (Gaebel, *J. Chem. Soc.*, 1911, Abstr. II., 448) is misleading. It is true that a slight mirror is obtained from relatively very large amounts of salvarsan, but the test is unsatisfactory if performed direct—*i.e.*, without previously decomposing with nitric and sulphuric acids.

Salvarsan may be distinguished from inorganic arsenic compounds by the following tests (Gaebel): (1) Hydrogen sulphide gives no precipitate with salvarsan, even after previous boiling with hydrochloric acid. (2) Bettendorf's reagent (solution of stannous chloride in hydrochloric acid) gives a yellow precipitate, soluble on warming, and reappearing on cooling. (With inorganic arsenic the reagent gives a brown coloration or brown precipitate.)

The organic part of the salvarsan molecule may be detected by colour reactions given by the diazo-derivative with (1) α-naphthylamine, or (2) resorcinol (Abelin, *Münch. Med. Wochenschr.*, 1910, **58**, 1002; see ANALYST, 1911, **36**, 452).

Neo-Salvarsan (or *Neo-Kharsivan*).—This compound was the result of an endeavour to obtain a drug similar to salvarsan in its physiological and therapeutic effects, and yielding a neutral solution. *Salvarsan* gives a solution acid in reaction, and before injection it is neutralised with sodium hydroxide, which throws down a precipitate; more sodium hydroxide is then added until the precipitate is just dissolved. The disadvantage of the alkaline solution prepared in this manner was eventually removed by the introduction of a methylene sulphoxy radicle into the molecule, resulting in the production of neo-salvarsan ("914"), having the formula



Neo-salvarsan is a yellow powder readily soluble in water, forming a neutral solution. Its physiological and therapeutic effects are similar to those of salvarsan. Owing to the introduction of the further group into the molecule, its arsenic content is two-thirds that of salvarsan; the corresponding dose of neo-salvarsan (having regard to the arsenic) is therefore one and a half times that of salvarsan.

Excretion and Secretion.—Salvarsan and neo-salvarsan are excreted fairly rapidly at first, the amount excreted *per diem* falling, as a rule, rapidly after about the second day, until at the end of about a week the quantity excreted is comparatively small (though the salvarsan may possibly still be detected by the α-naphthylamine or resorcinol test); arsenic may still be found in appreciable amount in the urine at the end of fourteen days, and, as a rule, can be detected at the end of three weeks, or even longer.

The following analyses show that in some cases the daily quantity excreted is not a rapidly diminishing one (Cases III. and IV., in which at the end of a week relatively large amounts of arsenic were still being passed).

In each of the following tables of urine analyses, column 1 represents the mgrms. of arsenic (calculated as As_2O_3) present in 100 c.c. of urine, and column 2 the mgrms. of arsenic present in the total volume passed in twenty-four hours. The analyses were carried out in every case on the twenty-four hour specimen.

Case 1.—0·6 *grm. Salvarsan given.*

Number of Days after Injection.	(1) Mgrm. As_2O_3 in 100 C.C. Urine.	(2) Mgrms. As_2O_3 in Total Twenty-four Hour Quantity.
1	1·0	2·8
3	1·0	3·7
5	0·17	0·73
7	0·17	0·22
8	0·07	0·35

Case 2.—0·5 *grm. Salvarsan given.*

Number of Days after Injection.	(1)	(2)
2	0·66	9·0
4	0·30	0·69
7	0·30	0·51
10	0·15	0·93
13	0·03	0·21

Case 3.—0·5 *grm. Salvarsan given.*

Number of Days after Injection.	(1)	(2)
2	0·2	3·1
5	0·2	1·7
8	0·15	2·1
11	0·05	0·38

Case 4.—0·5 *grm. Salvarsan given.*

Number of Days after Injection.	(1)	(2)
2	0·4	2·1
4	0·2	1·4
7	0·2	1·9
10	0·15	0·47
13	0·10	0·91

Case 4 (*continued*).—*Another* 0·5 *grm. Salvarsan given.*

Number of Days after Injection.	(1)	(2)
4	0·4	2·6
7	0·4	2·5
10	0·2	0·40
13	0·1	0·44
16	0·03	0·16
21	0·025	0·16

Case 5.—0·5 *grm. Salvarsan given.*

Number of Days after Injection.	(1)	(2)
3	1·3	8·8
5	0·8	5·9
8	0·33	3·1
11	0·12	1·3

Case 6.—0·5 *grm. Salvarsan given.*

Number of Days after Injection.	(1)	(2)
2	1·3	8·3
4	0·4	1·3
7	0·16	0·7
10	0·11	1·1
13	0·025	0·15

Analyses of Abdominal Viscera, Muscle, and Blood.

In cases of death following the injection of salvarsan and neo-salvarsan, arsenic is found in relatively large amounts in the liver, kidneys, and spleen, and in small amount in the muscles and blood. It is to be noted that arsenic is entirely, or almost entirely, absent from the brain and nervous system (any effect, therefore, on the nervous system is not due to the presence of salvarsan as such).

Below are given the results of analyses of various organs in nine cases of death following salvarsan treatment.

Case 1.

Aged twelve. Two doses 0·3 grm. salvarsan (one week interval). Death eight days after second injection.

	Mgrms. Arsenic per 100 Grms.
Liver	2·5

Case 2.

Aged twenty-three. Two doses 0·4 grm. salvarsan (four days interval). Death three days after second injection.

	Mgrm. Arsenic per 100 Grms.
Liver	0·83

Case 3.

0·5 grm. salvarsan given. Death twenty-nine hours later.

	Mgrms. Arsenic per 100 Grms.
Liver	4·0
Brain	0·01

Case 4.

Aged thirty-six. Two doses 0·6 grm. salvarsan (two weeks interval). Death two days after second injection.

	Mgrms. Arsenic per 100 Grms.
Liver	1·1
Kidney	0·2
Spleen	2·0
Brain	None detected.
Spinal cord	,, ,,
Aorta	0·025
Stomach contents	0·08

Case 5.

Aged nineteen. 0·6 grm. salvarsan given. Death nine days later.

	Mgrms. Arsenic per 100 Grms.
Liver	0·16
Kidney	0·13
Spleen	0·17
Spinal cord	? Trace
Small intestine	0·1
Large intestine	0·11

Case 6.

0·9 grm. neo-salvarsan given. Death four and a half hours later.

	Mgrms. Arsenic per 100 Grms
Liver	0·8
Kidney	1·0
Brain	0·02
Blood	0·05
Muscle	0·06
Bone	0·07

Case 7.

Dog weighing 24 lb. 3 oz. 0·3 grm. neo-salvarsan given. Animal killed twenty-four hours later.

	Mgrm. Arsenic per 100 Grms.
Liver	1·0
Kidney	0·4
Brain	Trace only.
Blood	0·1
Muscle	0·08
Bone	Trace only.

Case 8.

Two doses 0·9 grm. neo-salvarsan given (one week interval). Death four days after second injection.

	Mgrms. Arsenic per 100 Grms.
Liver	0·2
Kidney	1·5
Suprarenal gland	? Trace
Brain	Trace only.
Blood	0·02
Muscle	0·03
Bone	Trace only.
Aorta	None detected.
Skin	,, ,,

This case is of special interest. It will be noted that the amount of arsenic found in the kidney was relatively very high. The patient when admitted to hospital was suffering from acute nephritis.

Case 9.

Aged three weeks. Three doses of 0·02, 0·04, and 0·04 grm. respectively of salvarsan were given, with intervals of two days and one day. Death on day following last injection.

	Mgrms. Arsenic per 100 Grms.
Liver	0·4
Kidney	0·49
Spleen	0·39
Suprarenal gland	0·17
Lung	0·03
Brain	None detected.

PRESENCE OF SALVARSAN IN THE BLOOD AFTER INJECTION.

It has been stated (McIntosh and Fildes, *Proc. Roy. Soc.*, B, 1914, **88**, 324) that the blood is practically free from arsenic two days after an injection of salvarsan.

An experiment which I carried out points to this not being so in every case.

I have examined blood removed from a patient twenty-four hours, forty-eight

hours, and five days, after intravenous injection of 0·9 grm. of neo-salvarsan, and found respectively $\frac{1}{5}$ mgrm., $\frac{1}{10}$ mgrm., and $\frac{1}{40}$ mgrm. of arsenic per 100 c.c. of blood.

With regard to the presence of arsenic in the blood after salvarsan injections, it is to be noted that the arsenic is confined entirely, or almost entirely, to the serum.

In one case examined after injection of 0·6 grm. of salvarsan the blood was drawn off at the end of one hour, and the clot separated from the serum.

The serum was found to contain 0·5 mgrm. of arsenic per 100 c.c. serum.

In the case of the clot, 28 grms. gave only a faint trace (less than $\frac{1}{300}$ mgrm.), this trace being possibly due to incomplete separation of the clot from the serum.

Therapeutic Influence of the Milk of Lactating Women after Treatment by Salvarsan or Neo-Salvarsan.

It had been observed by Taege, and later by Duhot, that beneficial results had apparently been obtained from the ingestion by a syphilitic child of milk from the mother who had undergone treatment by salvarsan. This could be explained either (1) by the effect of the presence of salvarsan in the milk, or (2) by antibodies present in the serum of the mother.

Experiments carried out on human milk in two cases of salvarsan treatment point strongly to the latter view.

Case 1.

The mother received three injections of 0·4, 0·5, and 0·6 grm. of salvarsan at intervals of three days and five further days. The child was suckled the whole time. All symptoms of syphilis in both mother and child were stated to have cleared up within five days of the second injection.

Specimens of milk were taken (1) seventy-two hours after the first injection and (2) twelve hours after the second injection.

The former showed a faint trace of arsenic (less than $\frac{1}{100}$ mgrm. per 100 c.c. milk); in the latter no trace of arsenic was detected.

Case 2.

The mother received an injection of 0·5 grm. of salvarsan.

Samples of milk taken five and a half, ten and a half, sixteen, thirty-six, and sixty hours respectively after the injection showed no trace of arsenic by the Marsh test.

In all the above analyses the method employed for the estimation of the arsenic was treatment with nitric and sulphuric acids and subsequent reduction by potassium meta-bisulphite. The resultant solution was then used in the "electrolytic Marsh" apparatus, and the mirror obtained compared with standard mirrors prepared from known quantities of arsenic. The whole of the arsenic found was considered to be derived from salvarsan or neo-salvarsan respectively.

In conclusion, I desire to express my thanks to Colonel W. H. Willcox, A.M.S., in whose laboratory at St. Mary's Hospital I have been enabled to conduct these analyses, and to whom I am indebted for the cases detailed above.

Discussion.

Mr. A. Chaston Chapman said that one would not expect substances having the constitution of salvarsan or neo-salvarsan to be directly reduced in the Marsh apparatus, though in the Reinsch test one would expect to get the arsenic deposited on the copper. He would be interested to hear the exact procedure adopted in determining such very small quantities of arsenic occurring in such considerable quantities of viscera as must necessarily have been dealt with. For instance, in Case 3 0·01 mgrm. of arsenic was found in the brain. Although brain tissue was perhaps not peculiarly difficult to oxidise, yet in treating such a large quantity with nitric acid and sulphuric acid it would be reasonable to expect a considerable error. In the case of the liver the oxidation difficulties presumably were still greater. According to his own experience of the oxidation of organic matters in general with nitric acid and sulphuric acid, very low results were apt to be obtained, and those who were concerned to any considerable extent with the estimation of small traces of arsenic considered it, as a rule, desirable to avoid the destruction of organic matter whenever possible, as in many cases it was, though of course in such cases as those now under consideration it obviously was not. With a lead or cadmium cathode the sensitiveness was greater, but still not so great as with zinc and hydrochloric acid, and still less than when the zinc had been coated with cadmium. He could quite understand that for some purposes extreme sensitiveness was, as Mr. Webster had said, not necessary, but when the quantities of arsenic were so small as in some of these cases, he should venture to think that it would be best to employ as delicate a method of estimation as might be possible. From a chemical point of view it would have been interesting if some more information could have been given as to the methods to be adopted for the detection of salvarsan and its discrimination from, say, arsenious oxide or other inorganic forms of arsenic. Tests based on diazotisation had been suggested, but it seemed uncertain whether the diazo compounds of salvarsan or neo-salvarsan gave rise to such characteristic colours as would enable them to be distinguished from certain other bodies that might be obtained on extraction with alcohol.

Mr. R. Bodmer said that he had had a good deal of experience of the estimation of small quantities of arsenic in toxicological cases, but he must say that the accurate estimation of such small quantities as 0·01 mgrm. in 100 grms. seemed to him a rather difficult matter. The extreme delicacy of the zinc and hydrochloric acid method was, of course, well known, but he had always found it difficult owing, on the one hand, to the possibility of the zinc being insensitive, and, on the other, to the difficulty of getting zinc and hydrochloric acid absolutely free from arsenic. Quite recently he had been obliged to reject three different samples of zinc supplied as arsenic-free by one of the largest chemical dealers, and they were now unable to supply any which could be depended on. The electrolytic method, even though it might be a little less sensitive, was more reliable for work in connection with legal cases. He should like to ask Mr. Chapman how he avoided the destruction of organic matter, and whether, in his opinion, minute traces of arsenic could be satisfactorily estimated if the organic matter were undestroyed.

Mr. CHAPMAN said that if the arsenic were present in the form of arsenious oxide, it did not matter very much what quantity of organic matter was present; in fact, a little organic matter would increase the sensitiveness of the test. On the other hand, if the arsenic were in a form like that of salvarsan, it was obviously necessary to break up the organic compound. In a certain class of compounds with which he happened to have had to deal, and which contained nitrogen as well as traces of arsenic, he had found that on distillation with hydrochloric acid and a little ferrous chloride the arsenic came over still combined with nitrogen, and it was very difficult to get it in an uncombined state. In such cases the results would be low, unless the zinc was exceedingly sensitive. To ensure this he invariably adopted the method, which he had worked out some years ago, of coating the zinc with cadmium. If this were done, the question of "insensitiveness" of zinc practically disappeared. As to hydrochloric acid, he had never found the slightest difficulty in getting acid that would run for the greater part of an hour without showing the faintest trace of arsenic.

Mr. W. PARTRIDGE asked whether Mr. Webster could say in what form the salvarsan would be excreted. In some of the cases in which death followed the administration of salvarsan there seemed to be some doubt as to whether it was actually due to liberation of arsenic in the tissues.

Mr. WEBSTER said that diazotisation tests were not absolutely distinctive for salvarsan. Atoxyl gave almost the same colour with α-naphthylamine as salvarsan. With β-naphthylamine, however, atoxyl gave a brilliant colour, while salvarsan gave practically none. With resorcinol a brilliant red colour was given by salvarsan after diazotisation. He had found urine a week after the injection of salvarsan to give an appreciable colour with resorcinol or α-naphthylamine, although there was no coloration given by the urine before the injection. Such tests, of course, were not very delicate; arsenic would be found in the viscera or urine a long time after the diazotisation test ceased to give a coloration. With regard to the electrolytic method, this was admittedly less sensitive than the zinc and hydrochloric acid method. With platinum electrodes, which he was in the habit of using, the limit of sensitiveness in the ordinary way appeared to be about $\frac{1}{500}$ mgrm.; with lead electrodes it was stated to be about $\frac{1}{1000}$ mgrm.

Mr. CHAPMAN remarked that $\frac{1}{500}$ mgrm. was about the safe limit with zinc and hydrochloric acid, and it would be risky to reckon on such a degree of sensitiveness in the case of the electrolytic method.

Mr. WEBSTER said that, in his experience at any rate, $\frac{1}{300}$ or $\frac{1}{400}$ mgrm. might be reckoned upon in the electrolytic method. As to the quantities of material used and method employed, from 30 to 50 grms. of material were treated with nitric acid and sulphuric acid. That some loss occurred must be admitted, but the conditions were kept as nearly as possible the same in all cases, and the results, though admittedly rather low, were valuable for purposes of comparison. With regard to the zinc and hydrochloric acid method, he could never rely upon the zinc being absolutely free from arsenic, and should not care to use the method for medico-legal cases. With regard to Mr. Partridge's question, he did not think it was definitely known in what way salvarsan acted. It might be mentioned, however, that both salvarsan and neo-

salvarsan must not be used unless perfectly fresh; there must be no delay in using the contents of a tube after it had once been opened; otherwise there was great risk of toxic action. With regard to the form in which it was excreted, as already stated in the paper, urine passed several days after injection of "salvarsan" gave the diazo- and resorcinol tests for the benzene nucleus, and presumably some, at any rate, of the salvarsan was excreted as such, arsenic still being found in appreciable amount also.

THE EFFECT OF FEEDING ON THE COMPOSITION OF MILK AND BUTTER: DRIED YEAST AND DECORTICATED COTTON MEAL.

By HAROLD T. CRANFIELD and MARGARET G. D. TAYLOR.

In continuation of the feeding experiments carried out at the Midland Agricultural and Dairy College during the past few years, one was commenced in October of last year in order to compare the feeding value of dried yeast with that of decorticated cotton meal with regard to milk production. Arrangements were made, as before, to note the effects of the feeding on the composition of milk and butter.

Dried yeast is a by-product of the manufacture of beer. It appears on the market in the form of light brown flakes, and is a very bulky substance. It has a rather bitter taste, due to the presence of a small proportion of hop resin, but this does not appear to be detrimental to its use as a food.

Dried yeast has been used extensively on the Continent—especially in Germany—for many years, but it only appeared on the English market a few months before the commencement of the war. Previous to this only comparatively small quantities of yeast had been used for feeding—in its wet state—for pigs, the remainder being sold for manurial purposes or thrown away.

The dried yeast and decorticated cotton meal used in this experiment gave the following results respectively on analysis:

	Dried Yeast.	Decorticated Cotton Meal.
Moisture	9·51	10·55
Oil	0·75	8·91
"Albuminoids"	49·53	34·65
Soluble carbohydrates ...	32·02	26·86
Fibre	0·00	13·28
Ash	8·19	5·75
	100·00	100·00
Total food units	157·7	135·8

Experimental Details.—Following the details of the previous experiments, two sets of cows were selected, four in each set, particular care being taken in the selection with regard to previous milk production (quantity and quality) and periods of lactation.

Feeding.—The experiment commenced on October 12, consequently the cows received the bulk of their food in stall, and were obtaining very little grass.

First Week (*October* 12 *to* 19).—In order to get accustomed to their food, both sets of cows were started on their full experimental ration (given below), but the results of this week were not included in the averages for the two particular foods.

Daily Ration per Cow.

Set A.	Set B.
Basal ration: 1 lb. Egyptian cotton cake.	Basal ration: 1 lb. Egyptian cotton cake.
Basal ration: 1 lb. bran.	Basal ration: 1 lb. bran.
Basal ration: Hay (chopped and long).	Basal ration: Hay (chopped and long).
Basal ration: Cabbages.	Basal ration: Cabbages.
3 lb. dried yeast.	3 lb. decorticated cotton meal.

Second and Third Weeks.—Rations were the same as above.

Fourth Week (*Transition Period*).—Set A gradually changed from the dried yeast to the decorticated cotton meal, and Set B *vice versa.*

Fifth and Sixth Weeks.—The cows comprising Set A received the basal ration plus 3 lb. decorticated cotton meal, while those in Set B were given the basal ration plus 3 lb. dried yeast.

Milk.

Proportionate samples were taken of successive night's and morning's milk from each set of cows, the percentage of fat being determined by the Gerber method and the proteins from the aldehyde figure.

Fat.—The percentage of fat varied from 3·1 to 4·6, and the following data were obtained:

Weekly Averages per Cent.

	1st Week.	2nd and 3rd Weeks.	4th Week.	5th and 6th Weeks.
Set A ...	3·70	3·71	3·86	3·85
Set B ...	3·55	3·60	3·86	3·84

Total Average Percentages.

Set A.	Set B.	From Dried Yeast.	From Cotton Meal.
3·78	3·72	3·77	3·72

It will be seen from the above figures that there is only a slight difference between the two sets of figures. The dried yeast gave a slightly higher percentage of fat, but the difference was very small (0·05 per cent.).

Protein.—The aldehyde figure was determined on each sample of milk obtained from the successive night's and morning's milk of each set of cows, and the percentage of protein calculated by the factor 0·171. The following figures were obtained:

Weekly Averages per Cent.

	1st Week.	2nd and 3rd Weeks.	4th Week.	5th and 6th Weeks.
Set A ...	3·62	3·60	3·59	3·40
Set B ...	3·59	3·46	3·51	3·32

Total Average Percentages.

Set A.	Set B.	From Dried Yeast.	From Cotton Meal.
3·53	3·44	3·46	3·43

Here again the differences are very small, but still in favour of the dried yeast.

Butter.

On alternate days an aliquot portion of the night's and morning's milk from each set of cows was separated, and the cream ripened with a starter and churned after twenty-four hours.

The following determinations were made on the butter fat:

Reichert-Meissl value.
Kirschner value.
Polenské value.
Refractometer figure.

Reichert-Meissl Value.—The following averages were obtained:

	1st Week.	2nd and 3rd Weeks.	4th Week.	5th and 6th Weeks.
Set A ...	27·02	26·87	27·54	26·53
Set B ...	28·60	28·99	27·90	28·61

Set A.	Set B.	From Dried Yeast.	From Cotton Meal.
26·91	28·60	27·74	27·76

We can conclude from the above figures that the feeding exerted no individual influence on this value.

Kirschner Value.—The following average figures were obtained:

	1st Week.	2nd and 3rd Weeks.	4th Week.	5th and 6th Weeks.
Set A ...	19·50	20·39	20·83	20·20
Set B ...	21·00	22·84	22·45	21·60

Set A.	Set B.	From Dried Yeast.	From Cotton Meal.
20·29	22·09	20·99	21·62

There is a slight increase (0·63) in this figure in favour of the cotton meal. It is, however, too small to indicate any appreciable difference in the composition of the butter fats.

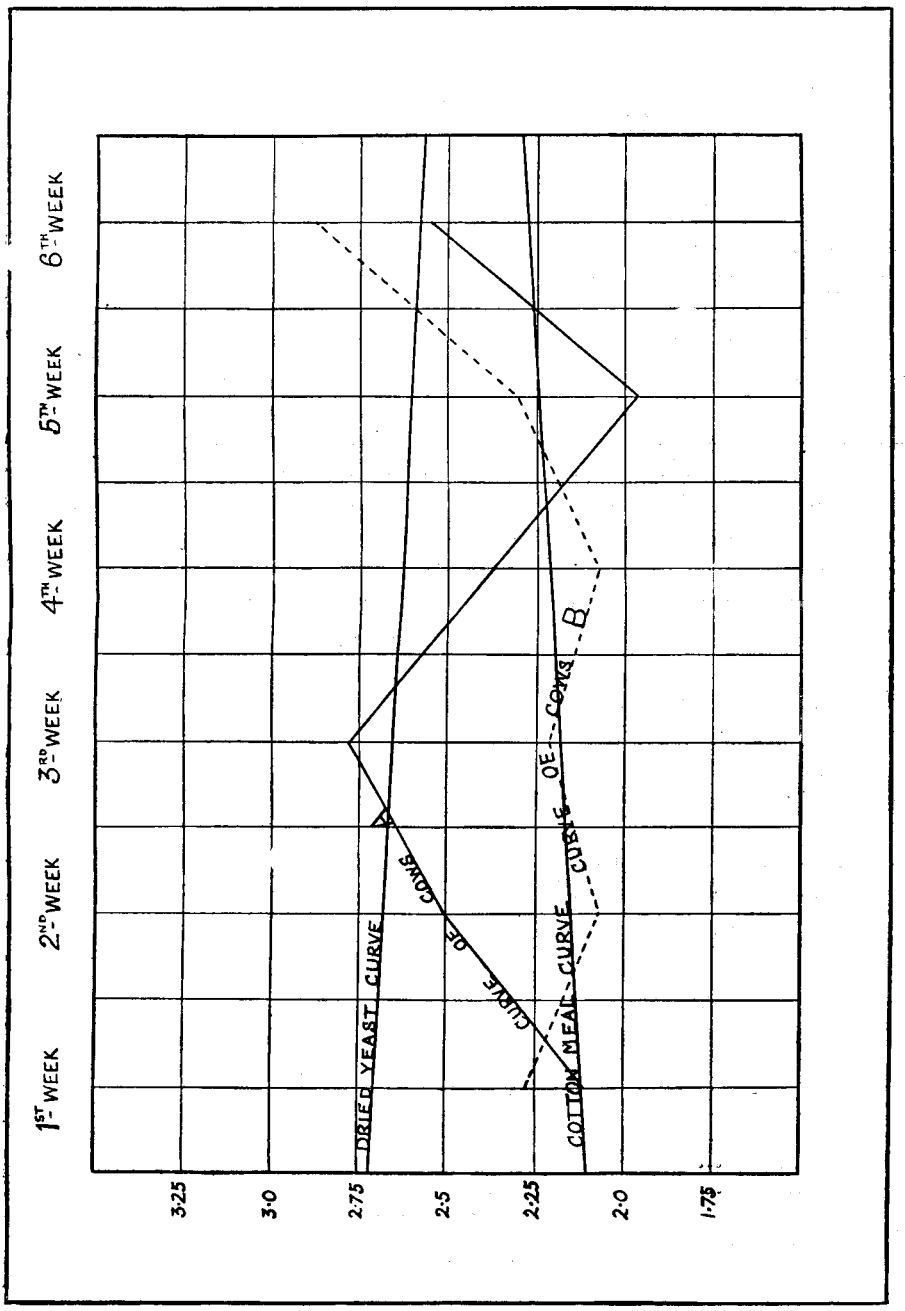
Polenské Value.—The following average figures were obtained:

	1st Week.	2nd and 3rd Weeks.	4th Week.	5th and 6th Weeks.
Set A ...	2·10	2·66	2·37	2·26
Set B ...	2·27	2·16	2·07	2·59

Set A.	Set B.	From Dried Yeast.	From Cotton Meal.
2·39	2·30	2·62	2·21

We find here a considerable variation due to the feeding, the dried yeast causing an

increase of 0·41 in the averages of this number. A graphical representation of these figures is shown in the following diagram :



POLENSKE VALUES.

Refractometer Figure.—Readings were taken at 35° C., using a Zeiss butyro refractometer. The average figures are as follows:

	1st Week.	2nd and 3rd Weeks.	4th Week.	5th and 6th Weeks.
Set A ...	47·25	46·68	46·37	46·29
Set B ...	46·67	46·36	46·37	45·82

Set A.	Set B.	From Dried Yeast.	From Cotton Meal.
46·57	46·23	46·25	46·32

There will be noticed a slight increase in favour of the cotton meal, but the variation (0·07) is too small to warrant further comment.

Quality of the Butter.—Practically no differences were noticeable between the two sets of samples of butter. Both were equally good as regards flavour and texture. The cotton meal butters gave, as a rule, a rather better colour than the dried yeast butters. No trace of a bitter flavour was noticed in the dried yeast samples.

Conclusions.

From these results it is evident that dried yeast is an excellent food for dairy cows as regards the quality of milk and butter. In spite of this substance being very low in oil content, the deficiency of oil in the ration does not appear to have influenced the quality of the milk or butter fat.

The only considerable variation in the composition of the fat occurred in the Polenské value, where the dried yeast feeding gave the higher figures.

We desire to tender our thanks to Miss B. Manners, N.D.D., for superintending the churning of the butter samples; and to Mr. John Dunlop, B.Sc., for facilities in the collection of the samples of milk.

Yield of Milk.

It has been suggested to us that it would be of interest to publish, in conjunction with this paper, the yields of milk obtained in this experiment. We have therefore approached Mr. John Dunlop, head of the Agricultural Department of Kingston College, and he has very kindly placed the necessary data at our disposal.

Weekly Milk Yields (in Pounds):

Set A.

	1st Week.	2nd Week.	3rd Week.	4th Week.	5th Week.	6th Week.
Cow No. 1	187¾	198½	194¼	187	165¼	156¾
,, No. 2	177¾	176¾	160¼	146¼	136	114
,, No. 3	193	195½	195½	170	179½	153½
,, No. 4	245½	250¼	231½	211½	190	176
Total weight of milk	804	821	781½	714¾	670¾	600¼
		Dried Yeast.			Decorticated Cotton Meal.	

SET B.

	1st Week.	2nd Week.	3rd Week.	4th Week.	5th Week.	6th Week.
Cow No. 5	168¾	169¼	175¾	162¾	164¾	143
,, No. 6	238½	228¼	205½	204¼	198¾	178¼
,, No. 7	178¼	183¼	151¾	101	126¼	125
,, No. 8	265¼	271¼	257½	234¼	216	200¼
Total weight of milk	850¾	852	790½	702¼	705¾	646½
		Decorticated Cotton Meal.			Dried Yeast.	

Total Weight of Milk.

	From Dried Yeast.	From Decorticated Cotton Meal.
Cow No. 1	392¾	322
,, No. 2	337	250
,, No. 3	391	333
,, No. 4	481¾	366
,, No. 5	307¾	345
,, No. 6	377	433¾
,, No. 7	251¼	335
,, No. 8	416¼	528¾
	2954¾	2913½

This indicates an increase of 41¼ lb. of milk in favour of feeding with dried yeast.

Since the average percentage of milk fat was 3·77 for the dried yeast, and 3·72 for the decorticated cotton meal, we get the following data:

Total Weight of Milk Fat.

From Dried Yeast.	From Decorticated Cotton Meal.
111·4 lb.	108·4 lb.

This shows an increase of 3 pounds of milk fat from feeding with dried yeast during the four weeks.

THE MIDLAND AGRICULTURAL AND DAIRY COLLEGE.

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ESTIMATION OF ACETONE IN PRESENCE OF ETHYL ALCOHOL.

BY JITENDRANATH RAKSHIT.

MESSINGER (*Ber.*, 1888, **21**, 3366) has described a titrimetric method of estimating acetone based upon its quantitative conversion into iodoform when acted on by caustic potash and iodine. Under these conditions some iodoform is formed from the ethyl alcohol, the amount of which depends on the temperature of the reaction. Although the substitution of ammonia for potash avoids the formation of iodoform from alcohol, its use is not practicable, owing to the decomposition of the iodides of

nitrogen into nitrogen and hydriodic acid and the consequent loss of free iodine. It was found that if potash be replaced by baryta or lime water, the estimation can be successfully carried out under the following conditions:

The sample to be examined, containing about 0·05 grm. of acetone, is placed in a 750 c.c. flask, and 300 c.c. freshly prepared lime-water added; the flask is loosely closed with a rubber cork, and heated to about 35° C. Drop by drop 5 c.c. of $\frac{N}{5}$ iodine solution are added and shaken for five minutes, then another 5 c.c. iodine solution is similarly added and shaken, and so on till 40 c.c. iodine solution are added. The gradual addition of iodine is necessary, because if all the iodine is added at once the reaction is not complete. If, during the addition of iodine, the colour persists after thorough shaking, more lime-water should be added. Ten minutes after the final addition of iodine a few drops of starch solution are added, the contents of the flask shaken and cooled, and 15 c.c. $\frac{N}{1}$ sulphuric acid added, and the excess of iodine titrated with $\frac{N}{10}$ sodium thiosulphate. The number of c.c. of $\frac{N}{5}$ iodine used up, multiplied by 0·00193, is the quantity of acetone in the sample taken. The results of some typical experiments carried out by the method are tabulated below:

Sample.	$\frac{N}{5}$ I consumed.	Acetone found.
	C.C.	Grm.
0·020 grm. acetone	10·2	0·0197
0·025 grm. acetone	12·8	0·0247
0·045 grm. acetone	23·3	0·0450
0·050 grm. acetone	25·7	0·0496
0·050 grm. acetone + 1 c.c. methyl alcohol	25·7	0·0496
0·050 grm. acetone + 0·125 c.c. ethyl alcohol ...	25·9	0·0500
0·050 grm. acetone + 0·50 c.c. ethyl alcohol ...	26·1	0·0503
0·050 grm. acetone + 0·75 c.c. ethyl alcohol ...	26·4	0·0509
0·050 grm. acetone + 1·0 c.c. ethyl alcohol	26·5	0·0510
0·050 grm. acetone + 2·5 c.c. ethyl alcohol	27·6	0·0532
0·050 grm. acetone + 4 c.c. ethyl alcohol	28·9	0·0557
0·050 grm. acetone + 5 c.c. ethyl alcohol	29·8	0·0575

It is thus seen that the error due to ethyl alcohol is slight. A mean of fifty analyses showed that 0·8 c.c. of $\frac{N}{5}$ iodine is absorbed by 1 c.c. of ethyl alcohol, and if this correction be applied to the last four analyses, the amounts of acetone become 0·0496, 0·0494, 0·0496, and 0·0498 grm. respectively. When the sample contains only about 1 part of acetone and 100 parts of ethyl alcohol, the results are not so reliable, as the correction figure becomes too high in comparison with that for acetone itself, but samples containing 1 part of acetone with 10 parts of alcohol give accurate and concordant results.

CHEMICAL LABORATORY,
GHAZIPORE, INDIA.

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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Estimation of Phosphorus in Plant Materials. A. W. Christie. (*J. Ind. and Eng. Chem.*, 1916, **8**, 511.)—The following methods for the oxidation of the organic matter were compared: Fusion with sodium peroxide; modified Neumann's method, consisting of digestion with sulphuric acid, 1 grm. of potassium sulphate and a drop of mercury; digestion with hot fuming nitric acid; and, finally, ignition with magnesium oxide. Hibbard (ANALYST, 1914, **39**, 100) describes this last method, but suggests "that some phosphorus may be lost from volatile organic substances containing it when ignited with magnesia." After the preliminary digestions the phosphorus was precipitated in the usual manner as ammonium phosphomolybdate, and finally weighed as magnesium pyrophosphate. The following table gives the values obtained, the author deciding in favour of the magnesium oxide method as being the quickest and most convenient:

	Per Cent. Phosphorus		
	Wheat Middlings.	Casein.	Lecithin.
Magnesia	0·56	0·86	3·58
Sodium peroxide	0·57	0·87	3·64
Modified Neumann method	0·57	0·86	3·61
Fuming nitric acid	0·38	0·65	1·58

H. F. E. H.

Gravimetric Estimation of Reducing Sugars in Cane Products. G. P. Meade and J. B. Harris. (*J. Ind. and Eng. Chem.*, 1916, **8**, 504-509.)—The Meissl and Hiller method was employed, in which 50 c.c. of the solution for analysis and 50 c.c. of mixed Fehling's solution are heated in a beaker to the boiling-point for four minutes, and maintained at gentle ebullition for exactly two minutes. At the end of the boiling period 100 c.c. of cold, recently boiled distilled water are added and the solution filtered through an alundum crucible or fused silica Gooch crucible. Normal lead acetate faintly acidified with acetic acid was used as a defecating agent. It was found that the results differed when the amounts of neutral lead acetate solution varied. Thus, 0·5 c.c. of lead solution freed from lead with sodium carbonate gave 0·2534 grm. of cupric oxide; whereas, when potassium oxalate was used, 0·2634 grm. was obtained, and 0·25 c.c. of lead solution gave 0·2657 grm. of cupric oxide after sodium carbonate, and 0·2715 grm.

after potassium oxalate. Carbonates, sulphates, and oxalates, are not interchangeable as de-leading agents, oxalates giving results from 4 to 5 per cent. higher on the weight of copper than when either of the others is used. Kieselguhr only, without the use of lead or other reagent, gives a clear filtrate both with final molasses and raw sugar, and the solution offers no mechanical difficulty in the precipitation and collection of the copper precipitate. Without lead the results are slightly lower than where lead and oxalate are used. The use of the thermometer to determine the beginning of the two-minute boiling period reduced the variations between the tests on the same solution. The copper should never be weighed as cuprous oxide, the results being about 5 per cent. higher on the weight of copper than those obtained by igniting to cupric oxide. Volumetric iodide determinations of the unreduced Fehling solution agree with the cupric oxide value. Under strict specifications as to the quantity and class of reagents, any method for the preparation of the solution for analysis will give results which agree well among themselves. Since many of the de-leading agents failed in their action, some estimations were made in which the excess of lead was left in the solution. The precipitation showed no abnormality, and the cuprous oxide was the usual colour and filtered without difficulty. The average weight of cupric oxide obtained from a molasses where no lead at all was employed was 0·2479 grm.; where lead and potassium oxalate were used, 0·2501 grm.; and where lead was used without removing the excess, 0·2451 grm. H. F. E. H.

Determination of the Gelatinising Temperature of Starches by Means of a Thermo-slide. C. K. Francis and O. C. Smith. (*J. Ind. and Eng. Chem.*, 1916, **8**, 509-511.)—An apparatus is described and illustrated by which water at a known temperature is circulated through a chamber containing a slide upon which is mounted in water the starch under examination. The temperature is noted when all the starch granules have lost their polarising properties, and this is taken as the true gelatinising temperature (*cf.* E. T. Reichert, *Pub. Carnegie Inst.*, 1913, p. 298). The thermo-slide method requires far less time than the water-bath method, and gives a very much sharper result. The following are some of the values obtained: Arrowroot, 74·6°; corn, 70·9°; navy bean, 75·6°; sweet potato, 82·4°; Irish potato, 67·8°; and wheat, 65·1° C. All these values are within 0·5° of those obtained by the water-bath method. H. F. E. H.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Digestibility of Bread. I. Salivary Digestion in Vitro. J. C. Blake. (*J. Amer. Chem. Soc.*, 1916, **38**, 1245-1260.)—Digestion experiments were carried out in presence of toluene on recently baked breads of varying composition, employing saliva, commercial ptyalin, Horlick's "Diastoid," and Taka-diastase. The materials were mixed at 25° C., and then kept in an incubator at 37° C., so as to simulate the conditions obtaining *in vivo*. Saliva was used in concentration of 1 in 8, the other three preparations in 1 per cent. solution. Portions for polarisation were pipetted

out at the requisite intervals, enzymic action being stopped by means of a 1 per cent. solution of arsenic acid. Three grms. of the inner part of the loaf cut into cubes were floated in 300 c.c. of the enzyme solution, the time being varied as required. The rate of digestion was followed by means of the polariscope, the only optically active substance entering the solution under the conditions described being maltose; it was noted, however, that dextrose appears in the digestions made with Taka-diastase. A number of dextrins were recognised and differentiated by the author, who also indicates the existence of two new ones. The three principal ingredients of cereal starches are stated to be amylocellulose composing the cell walls, amylopectin, and amylose. The amylose, contrary to the view of Maquenne and Roux (*Ann. Chim. Phys.*, [8], **9**, 179), was found to pass through the stages of amylodextrin and erythrodextrin during salivary digestion. All the dextrins, "under ordinary conditions," disappear from the solution within fifteen minutes, so that thereafter the further progress of the digestion can be followed by the polariscope, maltose being the only optically active sugar present, unless maltase has been added from some outside source. Maltose shows little muta-rotation, and "isomaltose, if present, has the same specific rotation as maltose." The amylocellulose (cell walls) digests only after more than twenty-four hours, and the only differences observable in the rate of digestion of bread made from hard or soft wheat and fermented more or less than usual were due to the relative amounts of gluten present. After the gluten was broken down, the rate of digestion was sensibly the same. It appears that under physiological conditions most of the amylose must be changed to dextrins in the mouth, and that these dextrins, together with most of the amylopectin and its hydrolytic products, are digested in the stomach, amylocellulose surviving until dealt with in the intestine. Stale bread digests very slowly unless its gluten be completely broken down. A considerable portion of the paper deals with a discussion of the identity of different "dextrins" and their iodine colorations. H. F. E. H.

Study of Plant Enzymes; particularly with Relation to Oxidation. (Test for distinguishing Dextrose and Lævulose.) A. D. Hall, E. F. and H. E. Armstrong, E. Keeble, and E. J. Russell. (*Report Brit. Assoc., Australia*, 1914, 108-109; through *J. Soc. Chem. Ind.*, 1916, **35**, 648.)—In certain white-flowered races of *Primula sinensis*, the oxydases (peroxydase) appear to be distributed in the flowers in definite zones, and it is probable that the zonal colour pattern in flowers of plants originating from crosses of white-flowered races of the plant with coloured forms is due to lack of uniformity in distribution of the peroxydase constituent of the colour-forming mechanism, not of the chromogen.

If a few drops of methylene blue be added to a freshly prepared solution containing 1 per cent. of dextrose or lævulose and 0·5 per cent. of sodium hydroxide solution, the blue colour is discharged almost immediately in presence of lævulose, but only after about fifteen minutes in presence of dextrose. After standing, the dextrose solution acts much more rapidly, whereas lævulose is less active than at first. The active agent is probably the enolic form common to both sugars, and the method may be used to compare the relative rates of enolisation of carbohydrates. Indigo-blue

solution, which changes from green to red, and finally to yellow, as it is reduced, may be used instead of methylene blue.

Urease has been detected in the root nodules from lupins and a number of other *Leguminosæ*, but attempts to detect it in organisms cultivated from the nodules gave negative results. The enzyme has also been detected by Benjamin in nodules from several Australian plants, including wattles, in tubercules derived from the Cycad *Macrozamia spiralis*, and in the seeds of *Abrus precatorius*.

ORGANIC ANALYSIS.

New Reaction of Aldehydes. R. de Fazi. (*Gazz. Chim. Ital.*, 1916, **46**, 334-359.)—On treating a few drops of a chloroform solution of an aromatic aldehyde with two or three drops of a 1 per cent. chloroform solution of acenaphthene and slowly adding 1 c.c. of strong sulphuric acid, a green ring changing to reddish-violet is obtained, or, if the tube be shaken, the sulphuric acid is coloured first green and then reddish-violet, the latter colour persisting for several days. The test is capable of detecting 0·0000078 grm. of benzaldehyde, 0·000019 grm. of vanillin, and 0·000006 grm. of furfural. It affords a means of distinguishing between aromatic and aliphatic aldehydes, which do not give these colorations. Formaldehyde for instance, gives a black precipitate which is insoluble in most organic solvents, while acetaldehyde yields a similar condensation product. The distinctive colorations (green changing to violet) are also obtained with aldoses and with carbohydrates capable of forming furfural or aromatic aldehydes on treatment with cold sulphuric acid. In the case of lactose, for example, an intense green coloration is produced in a few minutes, and this changes to violet after about forty minutes. Maltose reacts much more slowly than dextrose or lactose. C. A. M.

Estimation of Benzene and Toluene in Commercial Mixtures. A. Edwards. (*J. Soc. Chem. Ind.*, 1916, **35**, 587-590.)—According to the method proposed, the crude naphtha is separated by distillation into fractions each containing only two constituents, and the composition of the fractions estimated from their boiling-points, with reference to curves plotted from known mixtures. The correction of the thermometer readings by observations of the boiling-points of pure liquids is most important. For each thermometer a correction curve must be determined with pure hydrocarbons refractionated through a column into fractions boiling within a range of 0·2° C. The standard boiling-points of the pure liquids used for the correction of the thermometers are—Benzene, 80·2° C.; water, 100° C.; toluene, 110·7° C.; xylene, 139° C.; under a pressure of 760 mm. The variation of boiling-point due to changes in barometric pressure is 0·47° C. for every 10 mm., and is approximately the same for all three hydrocarbons. The separation of commercial distillates into fractions of two components is performed by distillation through a Young's twelve-bulb column, dividing the range from 80·2° to 139° C. into four portions, with re-fractionation of the two middle portions. In the case of "crude benzol" the presence of low-boiling constituents necessitates an additional separation up to 80·2° C. The fractions are dried by plaster of Paris, and the boiling-points determined in a special apparatus.

This consists of a round-bottomed flask with a cork carrying a reflux condenser and a short length of tubing $\frac{1}{2}$ inch in diameter reaching to a point just clear of the bottom of the flask. A thermometer is fixed in this tube so that its bulb is $\frac{1}{4}$ inch above the surface of the liquid. A hole is blown in the tube just below the cork to allow the vapour to circulate. The flask should be about half filled with liquid to the amount of 50-120 c.c. The liquid is caused to boil up the tube and over the bulb of the thermometer until the temperature is constant. The flame is then shifted slightly to one side so that ebullition takes place outside the tube, the temperatnre being read when it again becomes constant. The reading is corrected by the thermometer curve and for barometric pressure, and then referred to the boiling-point composition curve. For the analysis of crude products containing creosote, the sample is first rectified to 180° C. through a column, and the distillate again fractionated to 145° C. The product is washed with acid and alkali, dried and fractionated to 139° C. In such a distillate there is usually a considerable proportion of "forerunnings" boiling below 80·2° C. which would introduce an error in the benzene-toluene fraction. The separation is therefore extended to five fractions: (1) up to 85° C.; (2) 85° to 96° C.; (3) 96° to 110·7° C·; (4) 110·7° to 125° C.; (5) residue at 125° C. All the fractions except (2) and (5) are re-fractionated; from No. 1 all that comes over below 80·2° C. is collected separately; the residue goes to No. 2. Nos. 3 and 4 are fractionated to 110·7° C., the distillates going to No. 2 and the residues to No. 5; the boiling-points of these are then determined. The proportion of forerunnings collected separately is added to the benzene indicated by the curve; the toluene found will be exact, but the benzene will be high owing to the forerunnings. For the distillation of tar containing water the author describes a still of standard dimensions heated by an air-bath. The table from which the boiling-point composition curves are constructed is given below.

Boiling-Points of Mixtures of Toluene with Benzene and Xylene.

Toluene per Cent. by Volume.	Boiling-Points ° C.	
	Benzene and Toluene.	Xylene and Toluene.
0	80·2	139
10	81·8	134·4
20	83·7	130·3
30	85·75	126·7
40	88·1	123·5
50	90·75	120·8
60	93·8	118·4
70	97·2	116·3
80	101·0	114·3
90	105·3	112·5
100	110·7	110·7

J. F. B.

Improved Methods for Fat Analysis. E. B. Holland, J. C. Reed, and J. P. Buckley. (*Massachusetts Agric. Exp. Stat. Bull.* 166, 1915, 91-138.)—The bulletin deals, somewhat in detail, with the technique of the well-known methods of fat analysis. The large number of experiments on animal nutrition carried out at the Massachusetts Agricultural Experiment Station necessitated the analysis of many samples of fat, and in performing this work the authors have taken the opportunity to systematically study the methods commonly employed, and to embody in the report improvements suggested by foreign and American investigators.

The authors, though following to a great extent the ordinary procedure, have in most cases modified the reagents and the manipulation, and they call attention to the numerous precautions found necessary for accurate work. This is especially noticeable in the determination of insoluble acids, unsaponifiable matter, and iodine values.

Oils, fats, and waxes, are classified, and a number of useful tables are given, together with formulæ and other data of utility in carrying out the calculations connected with the determination of "constants." E. R. B.

Estimation of Small Quantities of Hydrocyanic Acid. M. O. Johnson. (*J. Amer. Chem. Soc.*, 1916, **38**, 1230-1235.)—The silver gravimetric method is not suitable for small amounts of cyanides, such as 1 mgrm., and the reducing substances in plant distillates interfere with this and the various titration methods. These reducing substances also interfere with the picric acid colorimetric method as shown by Chapman (ANALYST, 1910, **35**, 47; 1911, **36**, 269). The ferrocyanide method requires the cyanide solution to be concentrated to very small volume, and reagents must be added proportional to the amount of cyanide present to secure the maximum intensity of colour.

The author prefers the thiocyanate method, which he conducts as follows: The hydrocyanic acid is distilled into potassium hydroxide solution. To 50 c.c. of the distillate containing from 0·1 to 8 mgrms. of potassium cyanide, 1 c.c. of yellow ammonium sulphide is added, and the solution evaporated to dryness on the water-bath. The residue is extracted with three successive portions of 10 c.c. of acetone, which dissolves potassium thiocyanate, but not hydroxide or sulphide. The acetone extracts are evaporated to dryness and the cooled residue taken up in water and made up to 50 c.c. in a Nessler tube, to which 2 c.c. of 0·5 per cent. ferric chloride solution are added. The colour is finally matched with standards made by diluting various quantities of a solution containing potassium thiocyanate equivalent to 1 mgrm. of potassium cyanide per c.c., care being taken that precisely 2 c.c. of the ferric chloride solution are used in each experiment, as the intensity of colour developed depends to some extent on the concentration of ferric chloride.

If the distillate is coloured by organic matter and this colour passes into the acetone extract, the following procedure is adopted: The residue from the evaporation of the acetone extract is taken up in 25 c.c. of water, and the resulting solution is shaken in a separator with 25 c.c. of ethyl acetate, which will as a rule extract the yellow colour. The purified aqueous extract is then separated and the amount of cyanide determined as above described. G. C. J.

Estimation of Ferro- and Ferricyanides in the Presence of Cyanides and Thiocyanates. F. G. W. Knapman and E. L. Randall. (*Chem. News*, 1916, **113**, 265-266.)—The methods described depend on the reduction of ferricyanides by titanium trichloride. *Ferricyanides alone.*—The ferricyanide solution is diluted until it contains about 0·1 grm. per 50 c.c. It is then treated with an excess (about 2 grms.) of ammonium thiocyanate, and titrated with standardised titanium trichloride solution. The end-point of the titration is indicated by the abrupt formation of a golden-brown precipitate after the green colour of the mixture has gradually become fainter. *Ferrocyanides alone.*—The solution is acidified with a small quantity of sulphuric acid, dilute potassium permanganate solution is added until a red-coloured mixture is obtained, and, after the addition of an excess of ammonium thiocyanate, the solution is titrated with titanium trichloride solution. *Mixture of Ferrocyanide and Ferricyanide.*—A portion of the solution is titrated as described for ferricyanide, whilst another portion is oxidised with permanganate and the total ferricyanide then titrated. An excess of ammonium thiocyanate must be added in both cases. *Mixture of Ferrocyanide and Thiocyanate.*—In this case permanganate cannot be used to oxidise the ferrocyanide, since a white precipitate is formed which obscures the end-point. The solution should be treated with an excess of iodine solution, heated at 60° C. for about thirty minutes, the excess of iodine then destroyed by the addition of thiosulphate solution, using starch as indicator, and the mixture titrated with titanium trichloride solution after the addition of ammonium thiocyanate. *Mixture of Ferrocyanide, Ferricyanide, and Thiocyanate.*—The alkalinity due to cyanide is neutralised by the addition of dilute sulphuric acid, using phenolphthaleïn as indicator. A moderate excess of iodine solution is then added, and the solution then treated as described. The result gives the ferrocyanide *plus* the ferricyanide. The ferricyanide alone is estimated in a separate portion of the solution by neutralising the alkalinity, adding an excess of ammonium thiocyanate, and titrating with titanium trichloride solution. The methods yield accurate results, and may be applied to the estimation of ferrocyanide in "spent oxide." The presence of soluble chlorides and sulphates, provided these do not contain metals which yield insoluble double ferrocyanides, is without influence on the results.

W. P. S.

Estimation of the Methoxy Group in Compounds containing Sulphur. A. Kirpal and T. Bühn. (*Monatsh. Chem.*, 1915, **36**, 853-863; through *J. Chem. Soc.*, 1916, **110**, ii., 154-155.)—The authors describe improvements in their modification of the Zeisel method for the estimation of methoxy group. They find that it is not necessary to use hydrogen, and that a current of carbon dioxide gives satisfactory results. The pyridine to absorb the methyl iodide is now enclosed in two small bubbling tubes wholly composed of glass, the only necessary connection being for the attachment to the remainder of the apparatus. After evaporating off the pyridine in a dish, the remaining methiodide is dissolved in water and titrated with $\frac{N}{10}$ silver nitrate solution, using sodium chromate as an indicator. With this modification of the Zeisel process there is no need for a pure hydriodic acid, because the common impurities—for example, hydrogen sulphide and phosphine—do not affect the pyridine. The

new method is also applicable to the estimation of the methylimide group, but is of no value for the estimation of ethoxy group, as ethyl iodide is only partly absorbed by the pyridine. This method is also satisfactory for methoxyl estimations in sulphur compounds, but here the advantage over the Zeisel process is not so great as at first appears, because the latter can be made to give accurate results if the mixture of gas and vapour from the digestion flask is first passed through a slightly acidified solution of cadmium sulphate before reaching the silver nitrate solution. In this case any hydrogen sulphide is precipitated as cadmium sulphide. The unfavourable and inaccurate results obtained by the use of cadmium iodide solution are explained by the fact that with this reagent the cadmium sulphide produced tends to remain in part in colloidal solution, in which form it is more active and reacts with the methyl iodide, giving methyl mercaptan. Experimental results are given indicating the applicability of the Zeisel method with the use of cadmium sulphate to the estimation of methoxyl and ethoxyl groups in compounds containing sulphur in various states of combination.

Modification of Whipple's Method of Estimating Organic Nitrogen in Sewage. F. W. Bruckmiller and L. E. Jackson. (*J. Ind. and Eng. Chem.*, 1916, **8**, 499-500.)—The direct process for the estimation of the total nitrogen in sewage consists in the digestion of a known quantity of the sewage in acid solution, with or without copper sulphate, making decidedly alkaline with caustic soda, allowing to settle until clear, and then Nesslerising an aliquot portion. The following modification of Whipple's method (San. Res. Lab., **4**, 62) is recommended:

To 100 c.c. of sewage are added 5 c.c. of concentrated sulphuric acid and 2 c.c. of 10 per cent. copper sulphate solution. The mixture is digested until clear, and then for a further half-hour. A small crystal of potassium permanganate is added, and after cooling the whole is made up to 250 c.c.; 25 c.c. are pipetted out into a 100 c.c. Nessler tube; 25 c.c. of 5 per cent. caustic soda solution are added and the volume made up with ammonia-free water. The tube is then stoppered and allowed to stand twenty-four hours, after which time 10 c.c. are Nesslerised after diluting to a total volume of 50 c.c. It is found that the length of time the solutions are kept alkaline before Nesslerising influences the results, the best time being twenty-four hours, while increasing the concentration of potassium permanganate or copper sulphate had no effect. H. F. E. H.

Varnish Analysis. I. Molecular Weights of Vegetable Oils. M. Y. Seaton and G. B. Sawyer. (*J. Ind. and Eng. Chem.*, 1916, **8**, 490-493.)—The usual solvents for oils used in determinations of the molecular weight are not applicable to varnish analysis, since they give variable results, especially in the case of polymerised oils. For example, by the freezing-point method with benzene as solvent, the molecular weight of the fatty acids from raw linseed oil was fairly constant over a wide variation of concentration, but in the case of the fatty acids of a polymerised oil lowering of the molecular weight invariably accompanied an increase in the concentration. With nitrobenzene as solvent fairly accurate results were obtained, though excessive under-cooling caused some difficulty. Estimations by the boiling-point

method with chloroform or benzene as solvent also showed pronounced variations with the concentration, the lowering of the molecular weights increasing with the concentration. Satisfactory results were obtained by using stearic acid as the solvent, the ordinary commercial pressed material being quite suitable. In order to prevent the melted acid creeping up the side of the tube and stirrer, the top of the tube was surrounded by a piece of brass tube covered with asbestos paper on which was wound a resistance coil of nichrome wire. By passing a current through the wire the temperature of the tubing was kept at about 60° C., and the rising of the stearic acid checked. Prior to use the stearic acid was dried for some time in an oven below 100° C., and then did not readily absorb moisture from the air. To obtain concordant results it was necessary to keep the bath in which the freezing tube was immersed at approximately 40° C. Variations of more than 1° C. caused slight variations in the observed freezing-point. The following molecular weights calculated from the results show the accuracy and applicability of the method: Naphthalene (mol. weight 128), 126, 127·5; benzoic acid (mol. weight 122), 120-123·1; raw linseed oil (chemically refined), 740-760; refined linseed oil (heated to 600° F.), 735-765; refined linseed oil (heated one hour at 600° F.), 1000-1020; refined linseed oil (heated two hours at 600° F.), 1220-1250; (heated three hours at 600° F.), 1500-1530; fatty acids from refined linseed oil, 274-316; fatty acids from refined linseed oil (heated three hours at 600° F.), 335-380; raw Chinese wood oil, 825-840; polymerised Chinese wood oil (forty-five minutes at 450° F.), 1700-1760; soya bean oil, 719-750; polymerised Chinese wood oil, 1230-1250; and rosin, 282-288. C. A. M.

INORGANIC ANALYSIS.

Estimation of Carbonic Acid, Combined and Free, in Solution, particularly in Natural Waters. J. Johnston. (*J. Amer. Chem. Soc.*, 1916, **38**, 947-975.)—It is shown that the results of the ordinary titration method for free carbon dioxide cannot be interpreted unless the constitution of the solution is already known. Free carbon dioxide is readily determined by a gasometric method, provided that the concentration of combined carbon dioxide is very small, and even when it is large by observing precautions discussed in the text. The most common method of estimating the relative proportions of bicarbonate and carbonate—viz., by titration first with phenolphthaleïn and then with methyl orange—only yields approximate results, though results of sufficient accuracy can in many cases be obtained by following the procedure recommended by Küster (*Zeitsch. anorg. Chem.*, 1897, **13**, 127), and discussed in the present paper.

In general, however, it suffices to estimate (*a*) the total base equivalent to the combined carbonate, and (*b*) the total carbon dioxide, free and combined, both of which are easy, trustworthy, and unaffected by the presence of alkaline earths or iron. With a knowledge of these one can calculate (*c*) carbonate, (*d*) bicarbonate, and (*e*) hydrogen ion concentration, which is a measure of the acidity (or alkalinity) of the water; for, since we are dealing with an equilibrium capable of fairly rapid readjustment, we are justified in applying the equilibrium constants to calculate the above quantities in the great majority of cases in which a knowledge of them is of real importance. G. C. J.

Analysis of Acid Calcium Bisulphite Solutions. E. Hägglund. (*Chem. Zeit.*, 1916, **40**, 433-434; through *J. Soc. Chem. Ind.*, 1916, **35**, 686.)—The usual method employed for the analysis of the fresh "acid" in the pulp-mill is that of Winkler—viz., titration of the total sulphur dioxide by iodine, and of the "free" acid by sodium hydroxide in presence of phenolphthaleïn. In the latter, titration errors are caused by the presence of carbonic acid derived from the limestone and of volatile organic acids in the recovered gases blown off from the digesters. The end-point with phenolphthaleïn also is not sharp. Any satisfactory method must be readily applicable to mill routine, and must indicate the percentage of calcium oxide in the liquor with an error not exceeding −10 per cent. or +5 per cent. It is more dangerous to employ too little than too much calcium oxide in the digestion. A method for which greater accuracy is claimed was proposed by Oman, consisting in precipitating the calcium as normal sulphite by adding to 100 c.c. of the "acid" 30 c.c. of strong ammonia solution, filtering after fifteen minutes, and washing the precipitate three times with 50 c.c. of 15 per cent. ammonia each time. The precipitate is transferred to a 500 c.c. flask acidified with 10 c.c. of strong hydrochloric acid, the solution made up to the mark and titrated into 10 c.c. of iodine solution. Owing to the lesser solubility of calcium sulphite, practically the whole of the calcium sulphate is precipitated as sulphite, and a correction may be applied for this. The author has compared the Winkler and Oman methods with the results obtained by standard analytical methods, and found that both are liable to equally large errors, both frequently giving too low results for calcium oxide. The Oman method offers no advantage; apparently the calcium sulphite tends to oxidise on the filter, and possibly sulphur dioxide escapes during the acidification. A modification of the method is therefore proposed: 10 c.c. of clear sulphite "acid" are added to about 50 c.c. of water and 3 c.c. of strong ammonia. The liquid is made up to 100 c.c., the precipitate allowed to settle for fifteen minutes, and 10 c.c. of the clear supernatant solution drawn off and titrated with iodine. A correction based on the average percentage of calcium sulphate in the liquors, $= +0{\cdot}13$ per cent. SO_2, is applied. The maximum divergences recorded in a series of twelve analyses were equivalent to -1 and $+4{\cdot}1$ per cent. of CaO.

Rapid Method for Comparing the Decolorising Efficiency of Charcoals. L. Wickenden and J. W. Hassler. (*J. Ind. and Eng. Chem.*, 1916, **8**, 518-519.)—One hundred c.c. of a solution of Aniline Red, Soudan III., or of Oil Red RN, in kerosene oil (0·25 grm. per litre), are mixed with 1 grm. of a charcoal of average quality, and the beaker heated in a steam-bath. The contents are constantly stirred and filtered after ten minutes, and the colour of the filtrate given the arbitrary value 10. In like manner filtrates with 2, 3, 4 grms., etc., up to 10 are prepared, and numbered 20, 30, etc., up to 100. In testing a sample of charcoal, 5 grms. are treated with the aniline solution as described, and the colour of the filtrate compared with those of the standard filtrates. If, for example, the filtrate matches No. 70, 5 grms. of the charcoal will possess 40 per cent. more efficiency than an average charcoal. Charcoals which show high decolorising efficiency in this test show equally high results as decolorisers of cottonseed, coconut, and palm kernel oils. The solution

of the dyestuff can be kept for several weeks without perceptible fading, but it is advisable to test it at intervals with charcoal selected as the average standard.

C. A. M.

Separation of Erbium from Yttrium. P. S. Willand and C. James. (*J. Amer. Chem. Soc.*, 1916, **38**, 1198-1202.)—Experiments are described which had for their object the discovery of the best reagent for the fractional separation of erbium from a larger quantity of yttrium. The best reagents, among those tried, were sodium nitrite and potassium cobalticyanide.

When using sodium nitrite, the oxides are dissolved in hydrochloric acid, the solution is diluted and stirred with steam. Sodium nitrite solution in moderate quantity is then added drop by drop and the precipitate filtered off. More nitrite is added to the filtrate and the process continued, the final filtrate being treated with concentrated oxalic acid to recover the earths not precipitated by nitrite. This final precipitate is freed from sodium after ignition by boiling with water. When 10 grms. of mixed oxides were treated in this way and separated into six fractions, the first corresponded to an oxide of a metal with an atomic weight of 116, and the last to an oxide of a metal with an atomic weight of 90.

The separation effected by cobalticyanide was about as good as with nitrite. Sodium phosphate was somewhat less satisfactory as a fractional precipitant, whilst the other methods tried, which included fractional crystallisation of the ammonium double sulphates, diphenylmonosulphonates, picrates, and precipitation by means of ferrocyanides, were much less satisfactory. G. C. J.

Estimation of Lead as Lead Sulphite. H. Pellet. (*Ann. Chim. anal.*, 1916, **21**, 114-116.)—The lead solution is rendered slightly alkaline with sodium hydroxide, then acidified with acetic acid, and treated with an excess of sulphur dioxide. The latter is most conveniently obtained from a siphon of the compressed gas. The precipitated lead sulphite is collected on a filter, washed, dried, and weighed. The weight obtained is multiplied by 0·721 to obtain the equivalent quantity of metallic lead. This method of estimating lead is particularly useful in the case of sugar solutions which have been clarified with lead acetate. It also affords a means of separating lead from other metals which are precipitated by hydrogen sulphide. W. P. S.

Behaviour of Metals towards Certain Acids containing Hydrogen Peroxide. E. Salkowski. (*Chem. Zeit.*, 1916, **40**, 448-449.)—Copper, bismuth, nickel, gold, platinum, and antimony, are dissolved by a mixture of hydrochloric acid (sp. gr. 1·125) and 30 per cent. hydrogen peroxide solution. Silver and mercury do not dissolve in the mixture, and lead is attacked only slightly owing to the formation of a protective coating of lead chloride. Dilute sulphuric acid containing hydrogen peroxide dissolves copper, silver, nickel, and bismuth, but not tin, lead, gold, platinum, or antimony. A mixture of acetic acid and hydrogen peroxide dissolves copper, silver, mercury, lead, and bismuth, but not tin, nickel, gold, or platinum. Aluminium is soluble in the three acids mentioned, and the presence of

hydrogen peroxide is without effect. The insolubility of mercury in the mixture of hydrochloric acid and hydrogen peroxide is difficult to explain, seeing that the solvent action of the mixture is that of chlorine itself. It cannot be due to the catalytic action of the metal on the peroxide, or gold and platinum would not be dissolved, nor to the reducing action of the hydrogen peroxide on mercury chloride. Although hydrogen peroxide reduces mercuric acetate to mercurous acetate, it has no action on mercuric chloride. W. P. S.

Titrations with Permanganate in Strongly Alkaline Solutions. B. Brauner. (*Zeitsch. anal. Chem.*, 1916, **55**, 225-267.)—Arsenious acid, in strongly alkaline solution, reduces permanganate to the manganic condition state, provided that an electrolyte such as potassium sulphate is present to cause the colloidal manganese hydroxide, $Mn(OH)_3$, to separate as a precipitate. If the manganese hydroxide remains in colloidal suspension, the reduction proceeds as far as the manganous state. When manganous salts are oxidised in alkaline solution, hydrated manganese dioxide is produced, both from the oxidised salt and the reduced permanganate. Thallous salts are oxidised quantitatively to thallic (Tl_2O_3) salts, and cerous to ceric salts. Selenious and tellurous acids are converted into selenic and telluric acids respectively, and ferrous into ferric salts. Partial oxidation takes place in the case of lead, the product formed containing rather more oxygen than corresponds with the mixture $PbO + PbO_2$. In slightly alkaline solution nickel is oxidised to $Ni_{10}O_{11}$, whilst in strongly alkaline solution the oxidation proceeds approximately to the formation of Ni_2O_3. An oxide, containing somewhat more oxygen than corresponds with the formula Co_2O_3, is formed when cobalt is oxidised with permanganate in strongly alkaline solution. W. P. S.

Separation of Metals of the Tin Group in Qualitative Analysis. J. M. Welch and H. C. P. Weber. (*J. Ind. and Eng. Chem.*, 1916, **38**, 1011-1016.)—The reduction of stannic compounds to stannous compounds by means of metallic lead can be carried out with sufficient ease and accuracy to make it available as a method for the detection of tin. These two observations are the basis of the method of analysis which follows.

The sulphides of the tin group are separated from those of the copper group by treatment with ammonium polysulphide to which about 5 per cent. of sodium hydroxide has been added. This mixture does not dissolve copper sulphide as ammonium polysulphide alone does, nor does it dissolve mercury sulphide as sodium sulphide alone does. The sulphides of the tin group are dried superficially by suction or by pressing between filter-paper, and are then heated with 10 c.c. of hydrochloric acid for ten minutes on the water-bath. The arsenic sulphide is filtered off, and the filtrate is diluted to about 70 c.c., heated, and saturated with hydrogen sulphide. Antimony, if present, begins to separate before tin, and can be recognised by its red colour. If this gives place to brown with further addition of hydrogen sulphide, tin is present, and with practice the approximate ratio of tin to antimony can be judged by the shade. The mixture is next boiled to expel excess of hydrogen sulphide, 5 c.c. of 3 per cent. hydrogen peroxide solution are added, and the solution heated until the

precipitate is redissolved. Oxalic acid (5 to 10 grms.) is added and hydrogen sulphide again passed through the hot solution, which is allowed to cool while it is being saturated. The antimony sulphide is filtered off, and a portion of the filtrate is boiled with 1-2 grms. of granulated test lead for two or three minutes. The solution is then chilled and filtered into a solution of mercuric chloride. In presence of tin, white mercurous chloride is formed. The method will detect as little as 0·5 mgrm. of tin.

G. C. J.

Estimation of Tin in Tin Ashes. N. Welwart. (*Chem. Zeit.*, 1916, **40**, 458-459.)—The method described consists essentially in separating the tin as metastannic acid, converting this into the sulphide, dissolving it in hydrochloric acid, reducing the solution with metallic antimony, and titrating the resulting stannous chloride with standard iodine solution. From 3 to 10 grms. of the sample are boiled with 30 to 100 c.c. of nitric acid (1:1) until nitrous fumes are no longer given off, the solution is then diluted with three times its volume of water, boiled for a further five minutes, and filtered. The insoluble portion is washed first with dilute nitric acid, then with water, dried, ignited in a porcelain crucible, and weighed. A weighed portion of about 0·3 grm. of this ignited residue is fused with a mixture of sodium carbonate and sulphur, the melt dissolved in dilute hydrochloric acid, the solution boiled until all hydrogen sulphide has been expelled, and then diluted with three times its volume of water. To the hot solution are added 25 c.c. of concentrated hydrochloric acid and 2·5 grms. of antimony powder, and the mixture is boiled for thirty minutes while a current of carbon dioxide is passed into it; the mixture is then cooled, the current of carbon dioxide being maintained, and titrated with $\frac{N}{10}$ iodine solution which has been standardised against pure tin. Lead, copper, antimony, and zinc, do not interfere with the estimation, but if large quantities of iron are present the sulphide melt must be dissolved in water and the solution filtered before hydrochloric acid is added.

W. P. S.

Volumetric Estimation of Tin by Potassium Iodate. G. S. Jamieson. (*J. Ind. and Eng. Chem.*, 1916, **8**, 500-502.)—In the absence of interfering substances stannous solutions can be titrated satisfactorily with potassium iodate. Among the substances that interfere are ferrous, cuprous, and antimonious salts and precipitated metals.

Pure stannous solutions are made so strongly acid with hydrochloric acid that the fully titrated solution will not contain less than 10 per cent. of hydrogen chloride. To the cooled solution, contained together with 6 c.c. chloroform in a stoppered bottle, a 0·9 per cent. solution of potassium iodate (1 c.c. = 0·01 grm. tin) is added rapidly while shaking the bottle so as to give the contents a gentle circular motion until the iodine colour, which gradually appears, has increased to the maximum amount. The stopper is then inserted and the solution well shaken. The titration is continued with thorough shaking of the closed bottle after each addition until the end-point (disappearance of the violet colour of the chloroform) is obtained. With pure stannous solutions the results are exact.

Stannic solutions are reduced by boiling with sheet nickel and at least half their

bulk of concentrated hydrochloric acid for about forty-five minutes. Towards the end a current of carbon dioxide is introduced into the flask, and under these conditions the solution is cooled almost to 0° C. The cooled solution is filtered through absorbent cotton, sand (1 inch), and more cotton supported on a perforated disc in a calcium chloride tube, the filtrate being received in a stoppered bottle containing 6 c.c. of chloroform. Other methods of filtration, which were tried, failed to remove finely divided nickel which interferes with the subsequent titration. The flask and filter are washed with dilute (1:1) hydrochloric acid, and the solution titrated as above described. From the consumption of iodate, a deduction has to be made before calculating the tin. This deduction, which approximates 0·5 c.c., is determined by means of a blank determination, a fresh blank being run whenever a new sheet of nickel is taken into use.

With solders and type metals, a 0·5 grm. sample is heated with 15 c.c. of sulphuric acid, until sulphur dioxide is no longer evolved. After cooling, 20 c.c. of water and 15 c.c. of hydrochloric acid are added, after which the tin is reduced and titrated as above described.

Bronzes are decomposed with nitric acid, and the metastannic acid filtered off through a Gooch crucible. The washed precipitate and asbestos are boiled with 15 c.c. of sulphuric acid for two or three minutes, the solution cooled, 20 c.c. water and 15 c.c. hydrochloric acid added, the asbestos filtered off on a Gooch crucible, and the filtrate reduced and titrated as above described. G. C. J.

Use of Titanium Trichloride in Volumetric Analysis. A. Monnier. (*Ann. Chim. anal.*, 1916, **21**, 109-113.)—Titanium trichloride solution may be used for the titration of ferric and cupric salts and chromates, methylene blue being used as the indicator except in the case of cupric salts where safranine or induline is used for the purpose. In the titration of ferric salts, the solution is acidified with hydrochloric acid, 4 drops of a 0·2 per cent. methylene blue solution are added, the mixture is boiled and titrated, in an atmosphere of carbon dioxide, with titanium trichloride solution which has been standardised against a known quantity of iron. The disappearance of the blue colour indicates the end-point of the titration. The small quantity of titanium trichloride required to decolorise the added indicator is estimated separately and deducted from the total quantity. A similar procedure is adopted in the case of cupric salts. The reason for using safranine or induline in this titration is that these substances are not reduced until all the copper has been reduced, whilst methylene blue is reduced before the copper. The end-point in the titration of chromates is quite sharp, provided that the solution is sufficiently dilute. Copper and iron in the same solution may be estimated as follows: An aliquot portion is titrated in the presence of methylene blue, whilst a second aliquot portion is titrated with safranine or induline as the indicator. The first titration gives the quantity of ferric iron, the second the ferric iron and copper together. For the estimation of titanium in ores, etc., the hydrochloric acid solution of the metals is reduced with zinc, filtered, the filtrate treated with an excess of standard ferric chloride solution, and this excess then titrated with titanium trichloride solution.

W. P. S.

Many of these details are to be found in the monograph entitled "New Reduction Methods in Volumetric Analysis." By E. Knecht and E. Hibbert. Longmans, Green and Co. London, 1910.—EDITOR.

Estimation of Vanadium by Cupferron. W. A. Turner. (*Chem. News*, 1916, **113**, 284-285.)—The vanadium in a metavanadate solution can be precipitated quantitatively by means of Baudisch's "cupferron" reagent (the ammonium salt of nitrosophenylhydroxylamine; *cf.* ANALYST, 1910, **35**, 78, 327, 453). A necessary condition is that the solution be acid with a mineral acid, but not more than 1 per cent. of hydrochloric or sulphuric acid is needed. The 6 per cent. solution of the reagent, prepared as directed by Baudisch (ANALYST, *loc. cit.*), is added to the solution until the appearance of white nitrosophenylhydroxylamine shows that excess has been added, the vanadium compound being of a mahogany colour. A few c.c. of the reagent are added in excess, the precipitate is filtered off on paper without delay and washed with cold 1 per cent. sulphuric acid containing 1·5 grms. of "cupferron" per litre. The precipitate and paper are dried, then heated in a platinum crucible with a small flame to expel volatile substances, the paper incinerated at as low a temperature as possible, and the residue finally heated for some time in the uncovered crucible to insure its being in the highest state of oxidation (V_2O_5). The test numbers given in the paper are exact, but no example of separation of vanadium from other metals is given. G. C. J.

APPARATUS, ETC.

New Colorimeter. C. F. Sammet. (*J. Ind. and Eng. Chem.*, 1916, **8**, 519-521.)—The instrument described and illustrated in this paper is designed especially for determining the colour of commercial turpentines, but may serve for other purposes. Turpentines are now graded according to the depth of liquid required to match a Lovibond No. 1 yellow glass. Direct comparison is difficult, and the usual practice is to take as the standard a 25 mm. depth of the turpentine in question and a No. 2 yellow glass, and to compare this with a No. 1 yellow glass superimposed on a deeper layer of turpentine, the depth being varied until a match is secured. The depth in mm. of this second column of turpentine, less 25, is the depth corresponding to 1 Lovibond unit, and serves to grade the turpentine. The manipulation required by this method is tedious, and the eyes get so tired before the final comparison is made that only roughly approximate matches are secured.

The apparatus described and illustrated greatly simplifies the manipulation, and enables two observers to obtain closely concordant results. It consists essentially of a horizontal metal cell, 300 mm. long, and a vertical eyepiece mounted on a travelling carriage above the cell, the travelling carriage also carrying two 45° prisms, back to back and immersed in the liquid in the cell. The cell has glass ends, and each end is illuminated by a 100 watt metallic filament lamp of frosted glass, screened by a sheet of "daylight" glass. When the illumination at the two ends of the cell is equal and the eyepiece and prisms exactly in their middle position, the field observed through the eyepiece should appear uniform, whatever the contents of the cell. The cell is

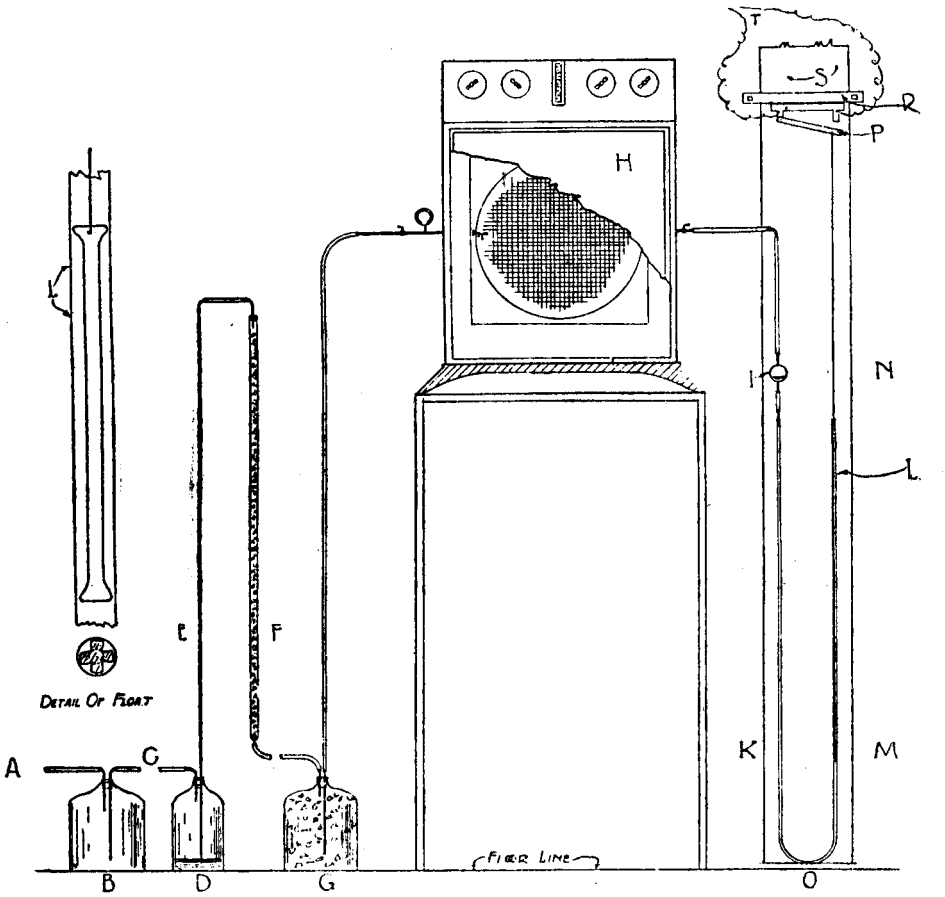
filled with a dark turpentine, and one of the lamps slightly shifted if necessary until the field appears quite uniform. A dark turpentine serves best for making this adjustment. The turpentine to be tested is now placed in the cell, a No. 1 yellow glass over one end of the cell, and a No. 2 yellow glass over the other end, and the carrier is moved towards the No. 2 glass until the field once more appears uniform. The difference between the distances from centre of the travelling carriage to the two ends of the cell gives the depth of turpentine corresponding to 1 Lovibond unit, and the carrier is provided with a pointer and the cell with a scale to enable these distances to be read off in mm. With this apparatus, two observers should never differ in their reports by more than 8 per cent., which is well within the limits required in grading turpentine. G. C. J.

Lutes and Cements. S. S. Sadtler. (*Chem. News*, 1916, **113**, 257-259.)—The following formulæ are given for the preparation of cements likely to be of use in the laboratory. *Water-proof.*—Various mixtures of asphalt, a solvent, and a filler, are described. Mixtures of boiled linseed oil, clay, asbestos, and red or white lead, are water-proof, as is also a mixture of flaxseed meal and water. Portland cement mixed with water containing a small quantity of sodium silicate or glue makes a good lute. *Oil-proof.*—(*a*) Glue, 2; glycerol, 1; water, 7, parts by weight. (*b*) Putty made of molasses and flour. (*c*) Glycerol, 90; water, 10, parts by volume; litharge, 90; red lead, 10, parts by weight. (*d*) Sodium silicate and whiting or barium sulphate. (*e*) Plaster of Paris and glue. *Acid-proof.*—(*a*) Many of the asphaltic mixtures are acid-proof. (*b*) Equal weights of rubber and boiled linseed oil, the rubber being first dissolved in carbon disulphide. (*c*) Rubber, 1; boiled linseed oil, 4; fire-clay, 6, parts by weight. (*d*) Rosin, 1; sulphur, 1; fire-clay, 2 parts, melted together. (*e*) Litharge, 80; red lead, 8; flock asbestos, 10 pounds, mixed with 6 quarts of boiled linseed oil. (*f*) Various mixtures of barium sulphate, powdered glass, china clay, casein, and sodium silicate. *Chlorine Resistant.*—Powdered glass, 1; Portland cement, 1; sodium silicate, 1. *Marine Glue.*—Crude rubber, 1; shellac, 2; pitch, 3, parts by weight, the rubber being first dissolved in carbon disulphide. *Machinists' Cement.*—Linseed oil, 6; rubber or guttapercha dissolved in carbon disulphide, 1 part, by weight; after twenty four hours, red lead is added in quantity sufficient to make a stiff paste. *Leather Cement.*—(*a*) Equal weights of glue and American isinglass, softened in water and then boiled with the addition of tannin. (*b*) Guttapercha, 1; benzene 10; linseed oil varnish, 12, parts by weight. (*c*) Guttapercha, 8; pitch, 1; shellac 1; and olive oil, 1; melted together. *Iron Cement.*—Iron filings, 40; manganese dioxide or flowers of sulphur, 10; ammonium chloride, 1; Portland cement, 20 to 40; and water sufficient to form a paste. *Crucible Cements.*—Mixtures of clay and borax, or sodium silicate and powdered glass and sand, may be used for cementing lids on crucibles. Fire-clay is the best cement for graphite, whilst tar or pitch forms an all-carbon binder. A water-proof cement which will stand a high temperature consists of a mixture of fine sand and 10 per cent. magnesium chloride solution made into a stiff paste, and painted with sodium silicate solution after application. *Magnesia Composition for Furnaces, etc.*—Hard burnt magnesia, 80, and light burnt magnesia,

20 parts, made into a stiff paste with water. *Oxychloride Cements.*—Magnesium oxide and wood pulp or sand are mixed with magnesium chloride solution (18° B.).

W. P. S.

Simple Device for Regulating Pump Used in Exhausting a Vacuum Oven. G. P. Plaisance and D. V. Moses. (*J. Amer. Chem. Soc.*, 1916, **38**, 1063-1065.)—Since even the best vacuum ovens are not air-tight, it is usual to operate the pump



continuously whilst the oven is in use; but, as the leakage is usually small, this is wasteful of power. The apparatus illustrated is intended to check this waste by cutting out the motor operating the pump when the vacuum is somewhat higher than is needed (say 20 mm. pressure, where an average of 40 mm. is desired), and switching on again whenever the vacuum falls below some approved minimum (*e.g.*, a pressure of 60 mm.). *A* is the tube leading to the pump, and *B* a trap to catch any mercury that may be drawn out of *D*. *D* contains mercury into which tube *E* (80 cm. long by 6 mm. internal diameter) dips to a depth of 1 cm. *F* and *G* contain calcium

chloride to prevent water given off by contents of oven from contaminating mercury or oil in pump. *H* is the oven. When the pump stops, mercury rises in *E* corresponding to the vacuum and closes the system. When the pump runs, the air has only to bubble through 1 cm. of mercury. Attached to the oven on the other side is the U-tube *KL* containing mercury and supported by standard *O*. *K* is 90 cm. long, and has an enlargement, *I* (3 cm. in diameter), 80 cm. from bend of U. *L* is 80 cm. long by 12 mm. internal diameter. It contains a glass float, *M*, 12 cm. long, with small bulb at base and small projections at top to centre it. The rod *N* connects *M* to an ordinary throw switch, *P*, on porcelain back *R*. The switch is mounted on a block, *S*, which can be slid up or down the standard *O*. The wires *T* lead to the motor. When the desired vacuum is reached, block *S* is adjusted so that float *M* will just open switch. As air leaks into the oven, the column of mercury raises float and closes the switch, thus starting pump. The use of the bulb *I* reduces the pressure required to throw the switch to 25 mm. of mercury, in place of 80 mm., the height which would be required were the tube without the bulb.

In the authors' experience with a Freas' electrically heated oven connected to a Crowell's pump, operated by a 1 horse-power motor, the device illustrated reduced the consumption of electrical energy over 80 per cent., the motor running for periods of about thirty seconds at intervals of three minutes or less. G. C. J.

REVIEW.

SOUTHALL'S MATERIA MEDICA. Revised and enlarged by ERNEST W. MANN, B.Sc. Pp. 244, with Glossary and Index. Price 7s. 6d. net.

This is the eighth edition of this work, now brought up to date with the new British Pharmacopœia.

It is frankly a compilation, and aims at presenting within a moderate compass the chief characters, important chemical properties, and practical uses, of the drugs of vegetable and animal origin, both official and unofficial.

It is divided into three sections, the first of which includes the organised drugs of vegetable origin, while the second deals with unorganised substances, such as juices, gums, resins, and volatile and fixed oils, and the third with the drugs which consist of animals, the parts of animals, and their derivatives.

In the first section the various substances are classified according to the parts of the plants from which they are obtained or of which they consist—seeds, roots, rhizomes, etc.—each having its allotted section, at the beginning of which is a short résumé of the general botanical and chemical characters of the group. The value of this arrangement is obvious, especially from the histological point of view. It would have been even of greater advantage had the scope of the work allowed the author to deal more thoroughly with the microscopical characters of the drugs dealt with, especially those that occur in the form of powder.

Brief mention is made of the forms of adulteration to which the various drugs are liable, and in the case of substances mentioned in the British, United States, and Indian Pharmacopœias the official descriptions and methods of analysis or standardisation (if any) are given in full.

In the case of the more out-of-the-way drugs there is a brief note on the important chemical and physical constants, where such are known.

The work is obviously intended mainly for the pharmacist, as the very large number of substances dealt with puts any extended reference to chemical matters out of the question. At the same time it contains information gathered from many sources about a number of drugs not often used in this country, and in the absence of larger works will be found a useful book of reference on the subjects with which it deals.

It is excellently and most systematically arranged, well and clearly printed, and full advantage has been taken of the use of varied type to distinguish and give prominence to official substances and preparations.

CECIL H. CRIBB.

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INSTITUTE OF CHEMISTRY.

PASS LIST: JULY (1916) EXAMINATIONS.

THE results of the Examinations of the Institute of Chemistry recently held in London have now been published.

Five candidates passed the Intermediate Examination—viz., A. C. Francis, Miss P. L. Garbutt, A. J. Hall, B.Sc. (Lond.), H. J. Hegan, B.Sc. (Lond.), and J. Sandilands. Nine candidates passed the Final (A.I.C.) Examination—viz.: In the Branch of Mineral Chemistry: E. G. Macintyre, B.Sc. (Glas.), and A. Stewart, B.Sc. (Glas.); in the Branch of Organic Chemistry: P. J. Brannigan, M.Sc. (Q.U.B.), T. Hopkins, B.Sc. (Lond. and Wales), D. Madden, A.R.C.S.I., James Ogilvie, B.Sc. (Edin.), and E. E. Wells, B.Sc. (Lond.); in the Branch of the Chemistry (and Microscopy) of Food and Drugs, Fertilisers and Feeding Stuffs, Soils and Water: H. E. Cox, B.Sc. (Lond.), and T. L. McEwan, B.Sc. (St. Andrews). One candidate examined for the Fellowship also took the Chemistry of Foods and Drugs, etc., and passed: J. Wood, M.A., B.Sc. (Aberd.).